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SPECIFICATION FOR *MAKHANNA* PRODUCTS

(First Reprint AUGUST 1986)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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September 1965

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SPECIFICATION FOR *MAKHANNA* PRODUCTS

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Indian Standard

SPECIFICATION FOR *MAKHANNA* PRODUCTS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 July 1965, after the draft finalized by the Edible Starches, Confectionery and Cereal Products Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 *MAKHANNA* is obtained by roasting *MAKHANNA* seeds which are obtained from the plant *Euryale ferox* Salisb. This plant is grown in ponds in some parts of Northern, Central and Western India. *MAKHANNA* is used in the preparation of *MAKHANNA* powder and fried *MAKHANNA*. These products have an export value and it is expected that the formulation of the specification would help in promoting the export of these products.

0.3 While formulating this standard, due consideration has been given to the relevant rules issued by the Government of India under the Prevention of Food Adulteration Act, 1954. This standard is, however, subject to the restrictions imposed under that act, wherever applicable.

0.4 This standard contains clause **4.1** which calls for an agreement between the purchaser and the vendor.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for *MAKHANNA*, *MAKHANNA* powder and fried *MAKHANNA*.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 *MAKHANNA* — The product obtained by roasting seeds of the plant

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*Rules for rounding off numerical values (*revised*).

Euryale ferox Salisb and removing the outer black covering as much as possible.

2.2 *MAKHANNA* **Powder** — The material prepared by grinding *MAKHANNA*.

2.3 Fried *MAKHANNA* — The product prepared by frying *MAKHANNA* in an edible frying medium. After frying, the material may be suitably salted, sweetened, spiced or otherwise flavoured.

3. REQUIREMENTS

3.1 *MAKHANNA* — The product shall be obtained by roasting *MAKHANNA* seeds. The outer black covering shall be removed as much as possible. If sand is used for roasting, care shall be taken to avoid contamination.

3.2 *MAKHANNA* **Powder** — The material shall be prepared by grinding *MAKHANNA*. It shall have a white to creamy colour and shall be free from extraneous matter and added colouring matter.

3.3 Fried *MAKHANNA* — The product shall be prepared by frying *MAKHANNA* in an edible frying medium and may be suitably salted, sweetened, spiced or otherwise flavoured. It shall be free from extraneous matter and added colouring matter. The material shall be crisp.

3.4 The products shall be manufactured in premises and equipment maintained under hygienic conditions (*see* IS : 2491-1963*).

3.5 The products shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in a suitable moistureproof container as agreed to between the purchaser and the vendor.

4.2 Marking — The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number, if any; and
d) Net weight of contents,

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*Code of sanitary conditions for food processing units.

TABLE 1 REQUIREMENTS FOR *MAKHANNA* PRODUCTS

(*Clause* 3.5)

SL No.	CHARACTERISTIC	REQUIREMENTS			METHOD OF TEST (REF TO APPENDIX)
		MAKHANNA	*MAKHANNA* Powder	Fried *MAKHANNA*	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture, percent by weight, *Max*	10·0	10·0	6·0	A
ii)	Total ash (on dry basis), percent by weight, *Max*	1·5	1·5	—	B
iii)	Total ash (on dry basis) (excluding common salt), percent by weight, *Max*	—	—	1·5	B
iv)	Acid insoluble ash (on dry basis), percent by weight, *Max*	0·1	0·1	0·1	C
v)	Crude fibre (on dry basis), percent by weight, *Max*	0·5	0·5	0·5	D
vi)	Crude protein content ($N \times 6.25$) (on dry basis), percent by weight, *Min*	8·0	8·0	—	E
vii)	Crude protein content ($N \times 6.25$) (on dry and fat free basis), percent by weight, *Min*	—	—	8·0	E

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix F.

6. TESTS

6.1 Tests shall be carried out as prescribed in the appropriate appendices given in col 6 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF MOISTURE

A-1. PROCEDURE

A-1.1 Weigh accurately about 5 g of the material (if the material is not in the powdered form, grind the material to a fine powder) in a dish made of porcelain, silica or platinum, previously dried in an air-oven and weighed. Place the dish in an air-oven maintained at 105° ± 2°C for five hours. Cool the dish in a desiccator and weigh with the lid on. Heat again at 105° ± 2°C in the air-oven for 30 minutes. Cool the dish in the desiccator and weigh. Repeat this process of heating for 30 minutes, cooling and weighing till the difference in weight between two successive weighings is less than one milligram. Note the lowest weight.

NOTE — Preserve the dish containing this dried material for the determination of total ash (**B-2.1**).

A-2. CALCULATION

A-2.1 Moisture, percent by weight

$$= \frac{100 (W_1 - W_2)}{W_1 - W}$$

where

W_1 = weight in g of the dish with the material before drying,

W_2 = weight in g of the dish with the material after drying, and

W = weight in g of the empty dish.

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*Specification for water, distilled quality (*revised*).

APPENDIX B

[*Table* 1, *Items* (ii & iii)]

DETERMINATION OF TOTAL ASH AND TOTAL ASH EXCLUDING SODIUM CHLORIDE

B-1. REAGENTS

B-1.1 Standard Silver Nitrate Solution — 0·1 N.

B-1.2 Standard Ammonium Thiocyanate Solution — standardized against standard silver nitrate solution.

B-1.3 Dilute Nitric Acid (1 : 9) — To 900 ml of water, add 100 ml of concentrated nitric acid (sp gr 1·42) and stir.

B-1.4 Nitric Acid (4 : 1) — To 100 ml of water, add 400 ml of concentrated nitric acid (sp gr 1·42). Bring the contents to the boil until a colourless solution is obtained.

B-1.5 Ferric Alum Indicator Solution — Prepare a saturated solution of ferric alum in water and filter.

B-2. PROCEDURE

B-2.1 Ignite the dried material (**A-1.1**) in the dish with the flame of a suitable burner for about one hour. Complete the ignition by keeping in a muffle furnace at 600° ± 20°C until grey ash results. Cool in a desiccator and weigh. Heat the dish again in the muffle furnace at 600° ± 20°C for 30 minutes and cool in the desiccator and weigh. Repeat this process of heating for 30 minutes, cooling and weighing until the difference between two successive weighings is less than one milligram. Note the lowest weight.

B-2.2 Dissolve the ash in 25 ml of the dilute nitric acid (1 : 9). Filter through Whatman filter paper No. 1 or its equivalent, collecting the filtrate in a 100-ml graduated flask, and wash the contents thoroughly with hot water. Make the volume to 100 ml.

B-2.3 To a 25-ml aliquot of the filtrate, add an excess of the standard silver nitrate solution (20 ml), stirring well to flocculate the precipitate of silver chloride. Filter and wash the precipitate thoroughly with water. To the combined filtrate, add 5 ml of each of the ferric alum indicator solution and concentrated nitric acid (4 : 1). Titrate the excess of silver nitrate with the standard ammonium thiocyanate solution to a stable light brown colour and point.

B-3. CALCULATION

B-3.1 Total ash (on dry basis), percent by weight $= \dfrac{100\,(W_2 - W)}{W_1 - W}$

where

W_2 = weight in g of the dish with ash (**B-2.1**),

W = weight in g of the empty dish (W under **A-2.1**), and

W_1 = weight in g of the dish with the dried material (W_2 under **A-2.1**)

B-3.2 Sodium chloride (on dry basis), percent by weight $= \dfrac{5{\cdot}85\,(V_1N_1 - V_2N_2)}{W_1 - W} \times \dfrac{V_3}{V_4}$

where

V_1 = volume in ml of the standard silver nitrate solution added initially,

N_1 = normality of the silver nitrate solution,

V_2 = volume in ml of the standard ammonium thiocyanate solution required for titrating the excess of silver nitrate,

N_2 = normality of the standard ammonium thiocyanate solution added,

W_1 = weight in g of the dish with the dried material (W_2 under **A-2.1**),

W = weight in g of the empty dish,

V_3 = volume in ml to which the filtrate was made up, and

V_4 = volume in ml of the aliquot of the filtrate taken for the experiment.

B-3.3 To obtain total ash excluding sodium chloride (on dry basis), percent by weight, deduct the value obtained under **B-3.2** from that obtained under **B-3.1**.

APPENDIX C

[*Table* 1, *Item* (iv)]

DETERMINATION OF ACID INSOLUBLE ASH

C-1. REAGENT

C-1.1 Dilute Hydrochloric Acid — approximately 5 N, prepared from concentrated hydrochloric acid (*see* IS : 265-1962*).

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*Specification for hydrochloric acid (*revised*).

C-2. PROCEDURE

C-2.1 Weigh accurately about 5 g of the material in a suitable dish made of porcelain, silica or platinum previously dried in an oven and weighed. Ignite the material with the flame of a suitable burner for about one hour. Complete the ignition by keeping in a muffle furnace at 600° ± 20°C until grey ash results. Cool in a desiccator and weigh. Heat again in the muffle furnace at 600° ± 20°C for half an hour and cool in the desiccator and weigh. Repeat this process of heating for 30 minutes, cooling and weighing till the difference in weight between two successive weighing is less than one milligram.

C-2.2 To the ash contained in the dish, add 25 ml of dilute hydrochloric acid, cover with a watch-glass and heat on a water-bath for 10 minutes. Allow to cool and filter the contents of the dish through Whatman filter paper No. 42 or its equivalent. Wash the filter paper with water till the washings are free from the acid and return it to the dish. Keep it in an electric air-oven maintained at 135° ± 20°C for about three hours. Ignite in a muffle furnace at 600° ± 20°C for one hour. Cool the dish in a desiccator and weigh. Repeat the process of igniting in the muffle furnace, cooling and weighing at half-hour intervals till the difference in weight between two successive weighings is less than one milligram. Note the lowest weight.

C-3. CALCULATION

C-3.1 Acid insoluble ash (on dry basis), percent by weight

$$= \frac{10\,000\,(W_2 - W)}{(W_1 - W)\,(100 - M)}$$

where

W_2 = weight in g of the dish with the acid insoluble ash;
W = weight in g of the empty dish;
W_1 = weight in g of the dish with the material; and
M = moisture, percent by weight, in the material (**A-2.1**).

APPENDIX D

[*Table* 1, *Item* (v)]

DETERMINATION OF CRUDE FIBRE

D-1. REAGENTS

D-1.1 Dilute Sulphuric Acid — 1·25 percent (*w/v*).

D-1.2 Sodium Hydroxide Solution — 1·25 percent (*w/v*).

D-1.3 Ethyl Alcohol — 95 percent by volume.

D-1.4 Ethyl Ether or Petroleum Ether — distilling below 65°C.

D-2. PROCEDURE

D-2.1 Dry to constant weight, about 5 g of the material in an electric oven maintained at 105° ± 1°C. Weigh accurately about 2·5 g of the dried material into a thimble and extract for about one hour with petroleum ether, using a Soxhlet apparatus. Transfer the fat-free material to a flask of one-litre capacity. Take 200 ml of dilute sulphuric acid in a beaker and bring to the boil. Transfer the whole of the boiling acid to the flask and immediately connect the flask with a water-cooled reflux condenser and heat so that the contents of the flask begin to boil within one minute. Rotate the flask frequently, taking care to keep the material from remaining on the sides of the flask and out of contact with the acid. Continue boiling for exactly 30 minutes. Remove the flask and filter through fine linen (about 18 threads to a centimetre) held in a funnel, and wash with boiling water until the washings are no longer acidic to litmus. Bring to the boil 200 ml of sodium hydroxide solution under a reflux condenser. Wash the residue on the linen into the flask containing the boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser and boil again for exactly 30 minutes. Remove the flask and immediately filter through the filtering cloth. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of ignited asbestos. Wash the residue thoroughly first with hot water and then with about 15 ml of ethyl alcohol. Dry the Gooch crucible and contents at 105° ± 2°C in an air-oven to constant weight. Cool and weigh. Incinerate the contents of the Gooch crucible in an electric muffle furnace at 600° ± 20°C until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

D-3. CALCULATION

D-3.1 Crude fibre (on dry basis), percent by weight

$$= \frac{10\,000\,(W_1 - W_2)}{W\,(100 - M)}$$

where

W_1 = weight in g of Gooch crucible and contents before ashing;

W_2 = weight in g of Gooch crucible and contents after ashing;

W = weight in g of the material; and

M = moisture, percent by weight, in the material (**A-2.1**).

APPENDIX E

[*Table* 1, *Items* (vi & vii)]

DETERMINATION OF CRUDE PROTEIN ON DRY BASIS AND ON DRY AND FAT FREE BASIS

E-1. APPARATUS

E-1.1 An assembly of the apparatus recommended for the determination of crude protein is shown in Fig. 1.

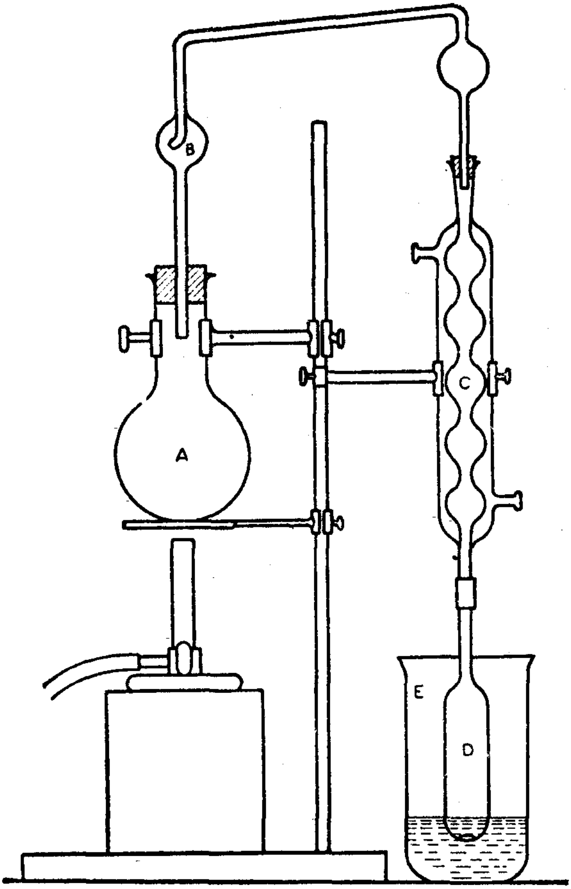

FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF CRUDE PROTEIN

E-1.1.1 *Description* — The apparatus consists of a round-bottomed flask *A* of 1 000-ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube *B*. The other end of the bulb tube *B* is connected to the condenser *C* which is attached by means of a rubber tube to a dip tube *D* open at the other end. The dip tube dips into the liquid contained in a beaker *E* of 250-ml capacity.

E-1.2 Soxhlet Extraction Apparatus

E-2. REAGENTS

E-2.1 Anhydrous Sodium Sulphate

E-2.2 Copper Sulphate

E-2.3 Concentrated Sulphuric Acid — sp gr 1·84.

E-2.4 Sodium Hydroxide Solution — Dissolve about 225 g of sodium hydroxide in 500 ml of water.

E-2.5 Standard Sulphuric Acid — 0·1 N.

E-2.6 Methyl Red Indicator Solution — Dissolve one gram of methyl red in 200 ml of rectified spirit (95 percent by volume).

E-2.7 Standard Sodium Hydroxide Solution — 0·1 N.

E-2.8 Ethyl Ether or Petroleum Ether — distilling below 65°C.

E-3. PROCEDURE

E-3.1 Transfer carefully about one gram of the material, accurately weighed, to a Kjeldahl flask, taking precaution to see that particles of the material do not stick to the neck of the flask. Add about 15 to 18 g of anhydrous sodium sulphate, about 0·2 g of copper sulphate and 25 ml of concentrated sulphuric acid. Place the flask in an inclined position. Heat below the boiling point of the acid until frothing ceases. Increase heat until the acid boils vigorously and digest for 30 minutes after the mixture becomes clear and pale-green or colourless. Wash down cautiously any particles sticking to the sides with a minimum quantity of concentrated sulphuric acid, and continue digestion for 60 to 90 minutes. Cool the contents of the flask. Transfer quantitatively, using water, to the round-bottomed flask *A* and dilute to 250 ml. Add a few pieces of pumice stone to prevent bumping. Then add about 60 ml of sodium hydroxide solution or more (if necessary, to make the solution alkaline) carefully through the side of the flask so that it does not mix at once with the acid solution but forms a layer below it. Assemble the apparatus as shown in Fig. 1, taking care that the dip tube *D* extends below the surface of a known quantity of standard sulphuric acid contained in the beaker *E*. Mix the contents of the flask by shaking and distil until all ammonia has passed over into the standard sulphuric acid. Detach flask *A* from the condenser and then shut off the burner. Rinse the condenser thoroughly with water into the beaker *E*. Wash the dip tube *D* carefully so that all traces of the condensate are transferred to the beaker. When all the washings have drained into the beaker, add two or three drops of methyl red indicator and titrate with the standard sodium hydroxide solution.

E-3.2 Carry out a blank experiment using all reagents in the same quantities but without the material to be tested.

E-3.3 In the case of fried *MAKHANNA*, take about 5 g of the material, grind it to powder, weigh accurately in a suitable thimble and extract with petroleum ether or ethyl ether for about 10 hours. Dry the extract contained in the Soxhlet flask, whose empty weight had been previously determined at 95° to 100°C, for 30 minutes. Cool in a desiccator and weigh. Continue the process of drying at 95° to 100°C for 30 minutes, cooling, drying and weighing until the loss in weight between two successive weighings is not more than one milligram. Record the lowest weight.

E-4. CALCULATION

E-4.1 Total protein (on dry basis), percent by weight

$$= \frac{875\,(B - A)\,N}{W\,(100 - M)}$$

where

B = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in the blank determination;

A = volume in ml of the standard sodium hydroxide solution used to neutralize the excess of acid in the test with the material;

N = normality of the standard sodium hydroxide solution;

W = weight in g of the material taken for the test; and

M = moisture, percent by weight, in the material (**A-2.1**).

E-4.2 Fat in fried *MAKHANNA* (on dry basis), percent by weight

$$= \frac{10\,000\,(W_1 - W_2)}{W\,(100 - M)}$$

where

W_1 = weight in g of the Soxhlet flask with the extracted fat;

W_2 = weight in g of the empty Soxhlet flask, clean and dry;

W = weight in g of the fried *MAKHANNA* taken (**E-3.3**); and

M = moisture, percent by weight, in the material (**A-2.1**).

E-4.3 Total protein (on dry and fat free basis), percent by weight

$$= \frac{87\,500\,(B - A)\,N}{W\,(100 - M)\,(100 - F)}$$

where

B = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in the blank determination;

A = volume in ml of the standard sodium hydroxide used to neutralize the excess of acid in the test with the material;

N = normality of the standard sodium hydroxide solution;

W = weight in g of the material taken for the test;

M = moisture, percent by weight, in the material (**A-2.1**); and

F = fat, percent by weight, in the material (**E-4.2**).

NOTE — Clauses **E-1.2**, **E-2.8**, **E-3.3**, **E-4.2**, and **E-4.3** are to be referred only in the case of fried *MAKHANNA*.

APPENDIX F

(*Clause* 5.1)

SAMPLING OF *MAKHANNA* PRODUCTS

F-1. GENERAL REQUIREMENTS OF SAMPLING

F-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

F-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

F-1.2 The sampling instrument shall be clean and dry when used.

F-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

F-1.4 The samples shall be placed in clean, odourless and dry glass containers or containers of any other suitable material. The sample containers shall be of such a size that they are almost completely filled by the sample.

F-1.5 Each container shall be sealed air-tight after filling and marked with full details of sampling, batch or code number, name of the manufacturer and other important particulars of the consignment.

F-1.6 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

F-1.7 Sampling shall be done by a person agreed to between the purchaser and the vendor and in the presence of the purchaser (or his representative) and the vendor (or his representative).

F-2. SCALE OF SAMPLING

F-2.1 Lot — All the containers in a consignment belonging to the same batch of manufacture shall constitute a lot.

F-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

F-2.2 The number of containers to be tested from a lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Total Number of Containers in the Lot (N) (1)	Number of Containers to be Selected (n) (2)
3 to 50	3
51 „ 200	4
201 „ 400	5
401 „ 650	6
651 and over	7

F-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them as 1, 2, 3,......... up to r, and so on in a systematic manner. Every rth container thus counted shall be chosen, r being the integral part of N/n, where N is the total number of containers in the lot and n the number of containers to be selected (Table 2).

F-3. TEST SAMPLE AND REFEREE SAMPLES

F-3.1 Preparation of Individual Samples — Empty out the contents of the container on a sheet of paper and mix thoroughly. Cone and quarter as often as necessary till about 200 g of the material is left. From this, take about 100 g of the material and divide into three equal parts. Each part so obtained shall constitute an individual sample representing the container and shall be transferred immediately to thoroughly clean and dry containers, sealed air-tight and labelled with the particulars given under **F-1.5.** The individual samples so obtained shall be divided into three sets in such a way that each set has a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the vendor and the third for the referee.

F-3.2 Preparation of a Composite Sample — From the material from each selected container remaining after the individual sample has been taken, equal quantities of material shall be taken and mixed together so as to form a composite sample weighing not less than 180 g. This composite sample shall be divided into three equal parts and transferred to clean dry containers made of glass and labelled with the particulars given in **F-1.5.** One of these composite samples shall be for the purchaser, another for the vendor and the third for the referee.

F-3.3 Referee Sample — Referee samples shall consist of a set of individual samples (**F-3.1**) and a composite sample (**F-3.2**) marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place agreed to between the two.

F-4. NUMBER OF TESTS

F-4.1 Test for description (**3.1, 3.2** or **3.3**) and crude protein estimation shall be conducted on each of the samples constituting a set of individual test samples (**F-3.1**).

F-4.2 Tests for the remaining characteristics, namely, moisture content, total ash, acid insoluble ash, and crude fibre shall be conducted on the composite sample as prepared under **F-3.2.**

F-5. CRITERIA FOR CONFORMITY

F-5.1 The lot shall be considered satisfactory in respect of the requirements tested in **F-4.1**, if each of the individual samples satisfies all the requirements.

F-5.2 The lot shall be considered satisfactory in respect of the requirements tested in **F-4.2**, if the test results on the composite sample satisfy the corresponding requirements.

F-5.3 The lot shall be declared to be in conformity with all the requirements of this specification, if it has been found satisfactory in **F-5.1** and **F-5.2.**